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THESIS

HOLOGRAPHIC INVESTIGATION OF
SOLID PROPELLANT PARTICULATES

by

Thomas R. Gillespie

December 1981

Thesis Advisor:

D. W. Netzer

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High speed, high resolution motion pictures were taken to compare the cinematic data with that available from the holograms.

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Holographic Investigation of Solid Propellant Particulates

by

Thomas R. Gillespie
Lieutenant Commander, United States Navy
B.S., U.S. Naval Academy, 1969

Submitted in partial fulfillment of the
requirements for the degree of

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December 1981

ABSTRACT

This investigation completed the development process to establish a technique to obtain holographic recordings of particulate behavior during the combustion process of solid propellants in a two-dimensional rocket motor. Holographic and photographic recordings were taken in a cross-flow environment using various compositions of metallized propellants. The reconstructed holograms are used to provide data on the behavior of aluminum/aluminum oxide particulates in a steady state combustion environment as a function of the initial aluminum size cast into the propellant.

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I. INTRODUCTION

The use of solid propellants in tactical missiles is widespread with a significant history of safety and reliability. The design and performance of the propulsion sections of these missiles are a function of the type of propellant used, its burning characteristics, and the use of any additives, among many others. Efforts to optimize these designs have led to the development of metallized propellants. These propellants generally employ finely powered aluminum (1-50 microns) in an attempt to capitalize on the conversion of its high heat of formation to kinetic energy for increased performance. In addition, these metallized fuels can provide a damping effect to potentially catastrophic combustion pressure oscillations within the combustor.

The addition of metal particulates introduces a new set of problems in the form of two phase flow losses as the combustion gases expand through the nozzle. These two phase flow losses arise from the presence of reacted aluminum oxide and from the presence of the products of the incomplete combustion of elemental aluminum. These particulates exist in the flow field in a range of mean diameters down to the submicron level. These solid particles cause a decrease in the flow exhaust velocity and consequently result in a reduction in thrust and specific impulse [Ref. 1]. Unburned aluminum also reduces the delivered specific impulse. In order to obtain the high theoretical performance available from these fuels, it is necessary to minimize these losses in practice.

Current solid rocket motor performance prediction models do not accurately address the behavior of these particulates due to a lack of experimental data in this area. Much quantitative data are needed which relate propellant properties and motor operating conditions (pressure, etc.) to the behavior of the particulates within the propellant port and through the nozzle.

Four experimental techniques are being used at the Naval Postgraduate School in an attempt to obtain this type of data. They are:

1. High speed cinematography of burning propellant strands in a combustion bomb and of burning propellant slabs in a cross-flow environment within a two-dimensional motor,
2. Residue collection and examination with a scanning electron microscope,
3. Scattered laser power spectra measurements to determine the mean particle diameter, and
4. Holographic images of propellant combustion in both strand and 2-D environments.

Diloreto [Ref. 2] conducted an initial investigation using high speed motion picture and electron microscope techniques.

Karagounis [Ref. 3] refined the motion picture technique for observation of propellant strands in a combustion bomb and developed a procedure for the holographic study of burning strands.

There are several points to be considered when using holographic techniques vice conventional photography. Utilization of the holographic procedure results in a film plate which has recorded on it both the phase and amplitude relationships of the received light, while a conventional

photographic procedure records only the amplitude. The unique recording obtained with the holographic technique results in a three dimensional image when properly reconstructed. Optimum utilization of the procedure can provide image resolution on the order of three microns [Ref. 4] with the flame envelopes surrounding the burning particles eliminated with narrow pass optical filters. Additionally, the depth of field for a holographic image has been determined to be 1.8mm for a 5 micron particle [Ref. 5] versus .018mm for a conventional photographic system with the same resolution [Ref. 6].

Sufficiently short pulse lengths (10 nsec) are available with Q-switched ruby lasers to record 5 micron particles moving at velocities up to 5000 cm/sec [Ref. 7]. If desired, double pulsing of the laser can provide multiple exposures of the same particles for determination of accurate velocity data.

The above capabilities of holography for the study of burning particulates are not without their price. Current data reduction techniques are slow and painstaking events, consuming many hours. Lasers with high power and short pulse lengths (<10 nsec) are currently limited to only a few pulses in a short period of time and therefore, only a small portion of the combustion process can be recorded.

This investigation was directed toward further application of the holographic method in order to study burning propellants in a 2-D slab motor where realistic cross-flow conditions can be simulated.

II. METHOD OF INVESTIGATION

Application was made of the holographic technique developed by Karagounis [Ref. 8] for the recording of the behavior of particulates from burning propellant strands in a combustion bomb. Modifications were made to the procedure to provide a higher laser power density, to reduce the recirculation of combustion products through the motor, and to reduce speckle during the reconstruction process. These changes were implemented to permit the recording of holographic images of propellant slabs, rather than strands, burning in a cross-flow environment.

High speed motion pictures were also taken to provide high resolution photographs for comparison with the corresponding hologram.

III. EXPERIMENTAL APPARATUS AND PROCEDURE

A. BACKGROUND

The utilization of laser technology was selected as the instrument of this study for a variety of reasons. As indicated earlier, the depth of field is an order of magnitude greater for a hologram as compared to a conventional photograph. Additionally, the use of a pulsed laser with an extremely short pulse length provides both the spatial and temporal coherence necessary to record high velocity particles without significant image blurring. Thomson [Ref. 9] recommends movement of less than one-tenth of a particle diameter during the length of the pulse to prevent blurring. The utilization of a laser capable of providing a high power level during a short pulse enables the use of a very fine grained recording medium with slow emulsions. Consequently, very high image resolution can be attained.

B. EXPERIMENTAL APPARATUS

The lens assisted holographic system used was provided by the U.S. Air Force Rocket Propulsion Laboratory, Edwards, California. It was used as described in the system operating manual [Ref. 10]. The distinguishing feature of this system is the design of the holocamera in which the AGFA-GEVAERT 8E75 HD film plate is mounted on a kinematic plate holder near the focal plane of a pair of plano convex lenses. This device serves to hold the plate during both recording and reconstruction of the image. A photograph of this system is shown in Figure 1.

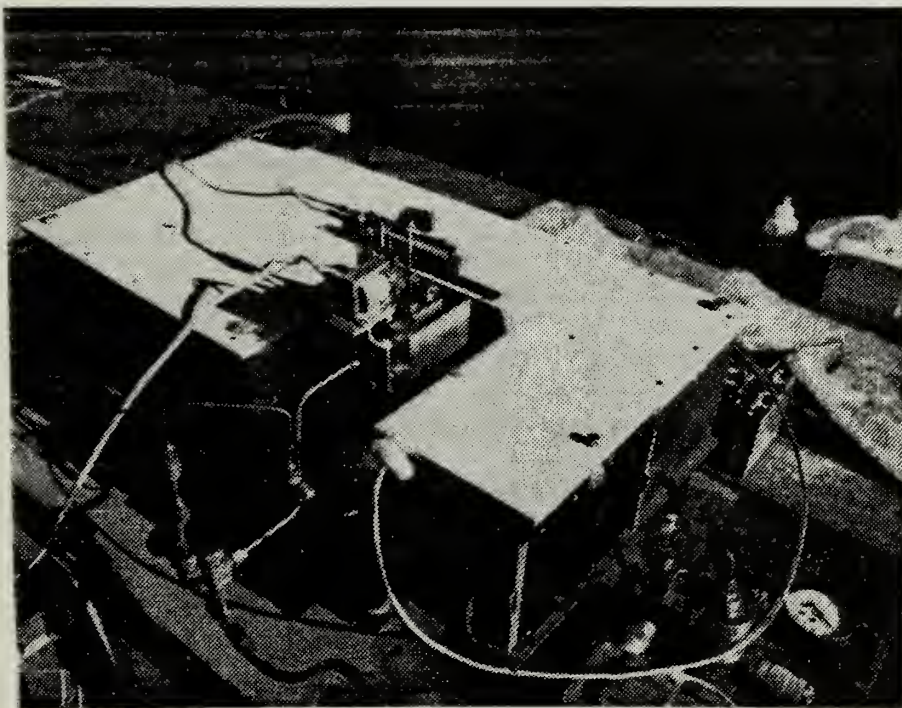


Figure 1. Lens Assisted Holographic System

A Q-switched pulsed ruby laser was used for recording holograms. It is described in detail in its instruction manual [Ref. 11]. The laser consists of a Q-switched oscillator, a ruby amplifier, a beam expanding telescope, an alignment autocollimator, a low power helium-neon pointing laser, and the associated power supplies. The laser operates at a wavelength of .6943 microns and is capable of producing a variety of pulse lengths including a 50 nanosecond and a 10 nanosecond pulse. Power levels of one joule per pulse are available down to the 50 nanosecond pulse. The power of the 10 nanosecond pulse is approximately one-fourth joule. Output beam diameter is a function of the focal length of the diverging lens in the beam expander. The output beam is currently about 1.25 inches in diameter. A photograph of this equipment is provided as Figure 2.

Image reconstruction was provided by a Spectra-Physics Model 125 He-Ne CW gas laser with an output of 80 milliwatts at .6328 microns. To view a hologram the film plate was rear illuminated by the reconstruction laser and the projected image was viewed with a variable power microscope. This method ensures that aberrations of the scene beam caused by the focusing lenses cancel as the image is projected back through the optical system and better resolution is obtained. Microphotographs of the projected holographic images were made on Kodak Ektachrome 64, 35mm slide film, using a Canon Model F-1 camera mounted on the microscope. The Canon Model F-1 allows for the use of various ground glass focusing plates. This permits optimum focusing of the image on the film plane for different light intensities from the hologram. The microscope and Kinematic Plate Holder were mounted on an optical bench to provide stability and to ensure consistency of results for each photograph. This apparatus was

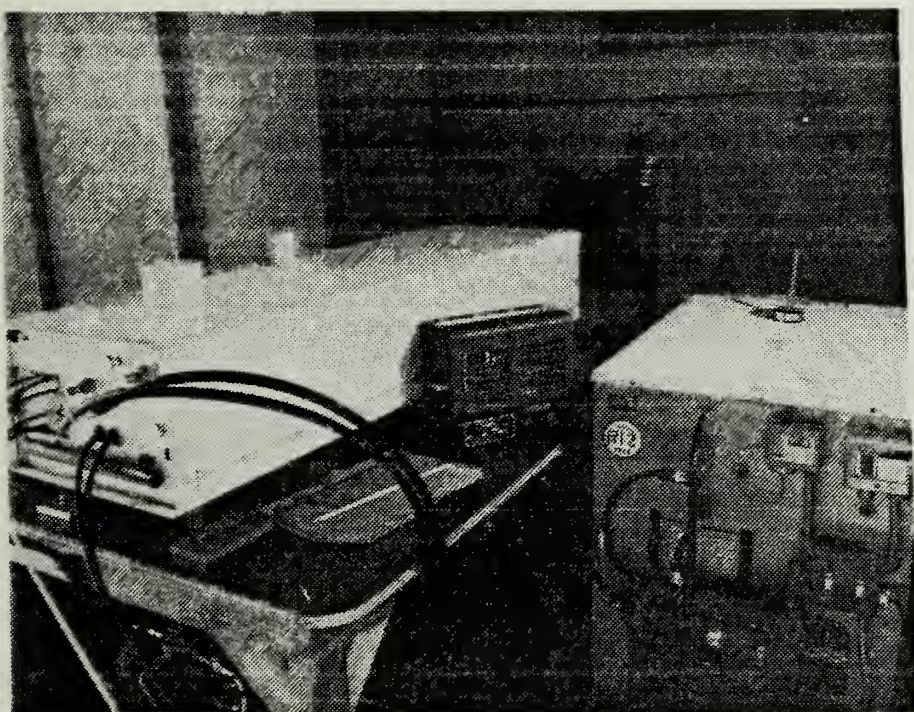


Figure 2. Q-Switched Pulsed Ruby Recording Laser

installed in a space with a filtered atmosphere to minimize dust induced interference in the reconstruction laser beam. This system is represented in Figure 3.

The two-dimensional motor was designed and built to receive opposing propellant slabs. Two 3/8 inch diameter high quality glass ports were provided to allow a field of view of the burning propellant. The ports were centered at the lengthwise midpoint of the propellant. The free volume of the motor was approximately 0.013 cubic inch and was adjustable by the removal or addition of spacers within the motor.

The spacers mounted on the window shutter blocks were constructed of plexiglas due to its ease of fabrication, availability in a variety of thicknesses, and it is electrically non-conductive and so helped prevent grounding of the propellant ignition system. In addition, a stainless steel L-shaped spacer was bonded to the face of one of the plexiglas spacers to seal the combustor and to promote cross flow velocity development as the propellant ignited. The other plexiglas spacer was shimmed to ensure that both sides of the propellant were in physical contact with the plexiglas. This tended to prevent uncontrolled burning, and with the steel spacer, helped to dampen the recirculation of the combustion products through the combustor. This is shown in Figure 4.

To protect the glass ports from fouling by combustion products before a hologram could be taken, a set of retractable window shutters was installed in addition to a nitrogen purge system. These shutters remained closed with the purge system operating until steady state conditions were reached. At that time, a combustion chamber pressure sensor triggered a solenoid, and via a time delay, released spring loaded

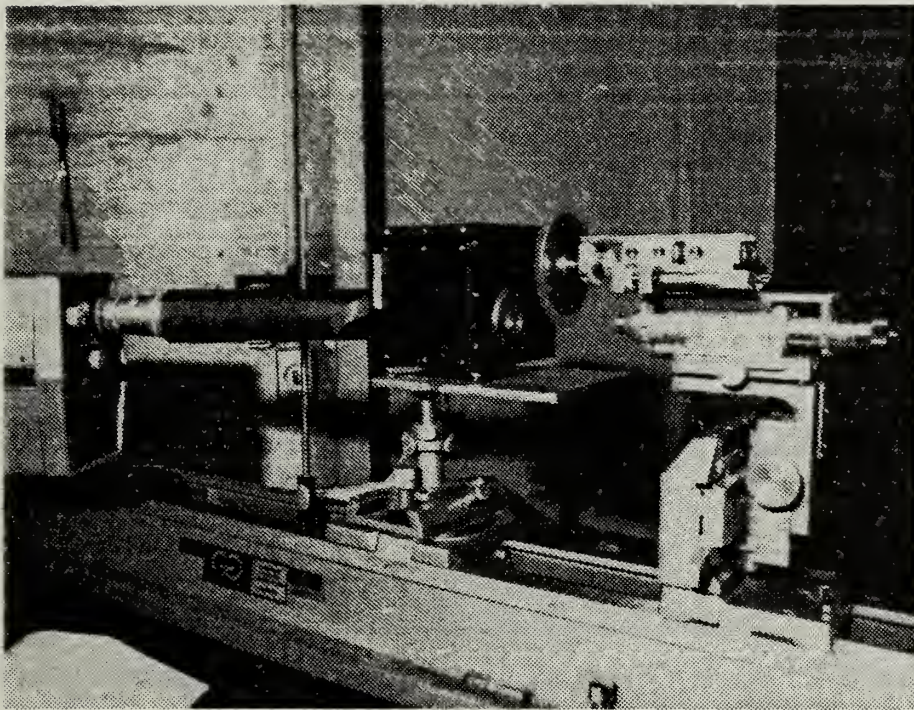


Figure 3. Holographic Reconstruction Apparatus

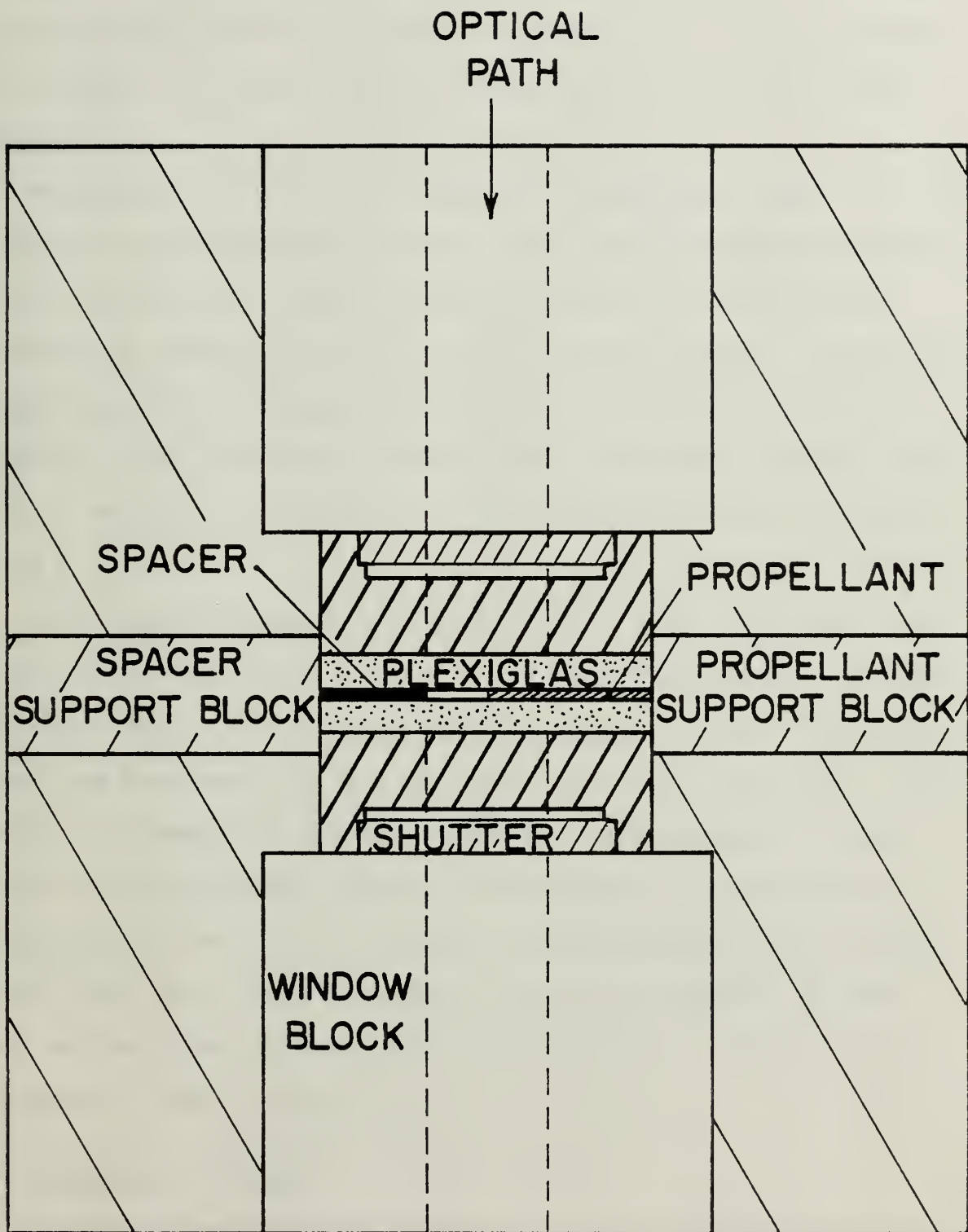


Figure 4. Plexiglas Spacer Arrangement

shutters. The rising shutters in turn tripped a microswitch at the end of their travel and fired the laser. Pressure-time plots were provided by a Honeywell Visicorder which also marked the various events as they happened.

Mounted atop the propellant chamber was an additional chamber for the introduction of pressurizing nitrogen. This chamber reduced the tendency of the pressurizing nitrogen to cause recirculation in the combustor, allowing the combustion products to clear the viewing area. A converging nozzle was also used between the two chambers to minimize recirculation effects. A .063 inch throat diameter graphite nozzle was secured to the exit of the pressurization chamber. This apparatus is shown in Figures 5 and 6.

A Red Lake Laboratories Hycam motion picture camera was used to record the high speed motion pictures of the burning propellant. The scene was back lighted by a 1200 watt Selectroslide Model SLM-1200 light source. Kodak 16mm Ektachrome 7239 film was used at f1.9 for all exposures. After developing, these films were used to provide a two dimensional dynamic view of the corresponding holographic representation and preliminary timing data for the initial holograms for each propellant sample. The movies could not be taken simultaneously with the holograms, but they were recorded under the same pressure-time parameters. The system arrangement is shown in Figure 7.

C. EXPERIMENTAL TECHNIQUE

Holographic recordings began with the preparation of propellant samples. Propellant composition and initial particle size are listed in Table 1.

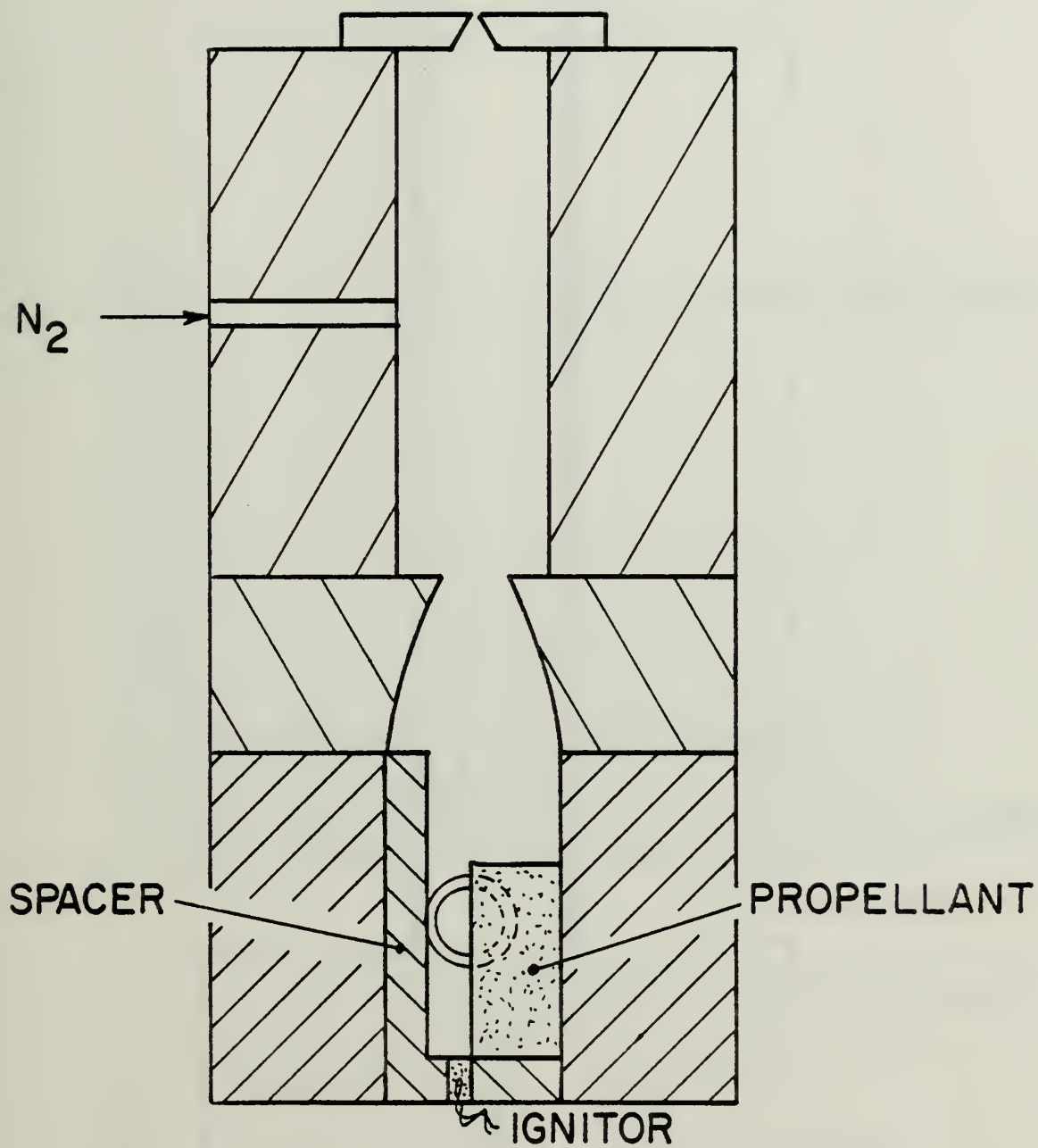


Figure 5. Motor Line Drawing, Window Side View

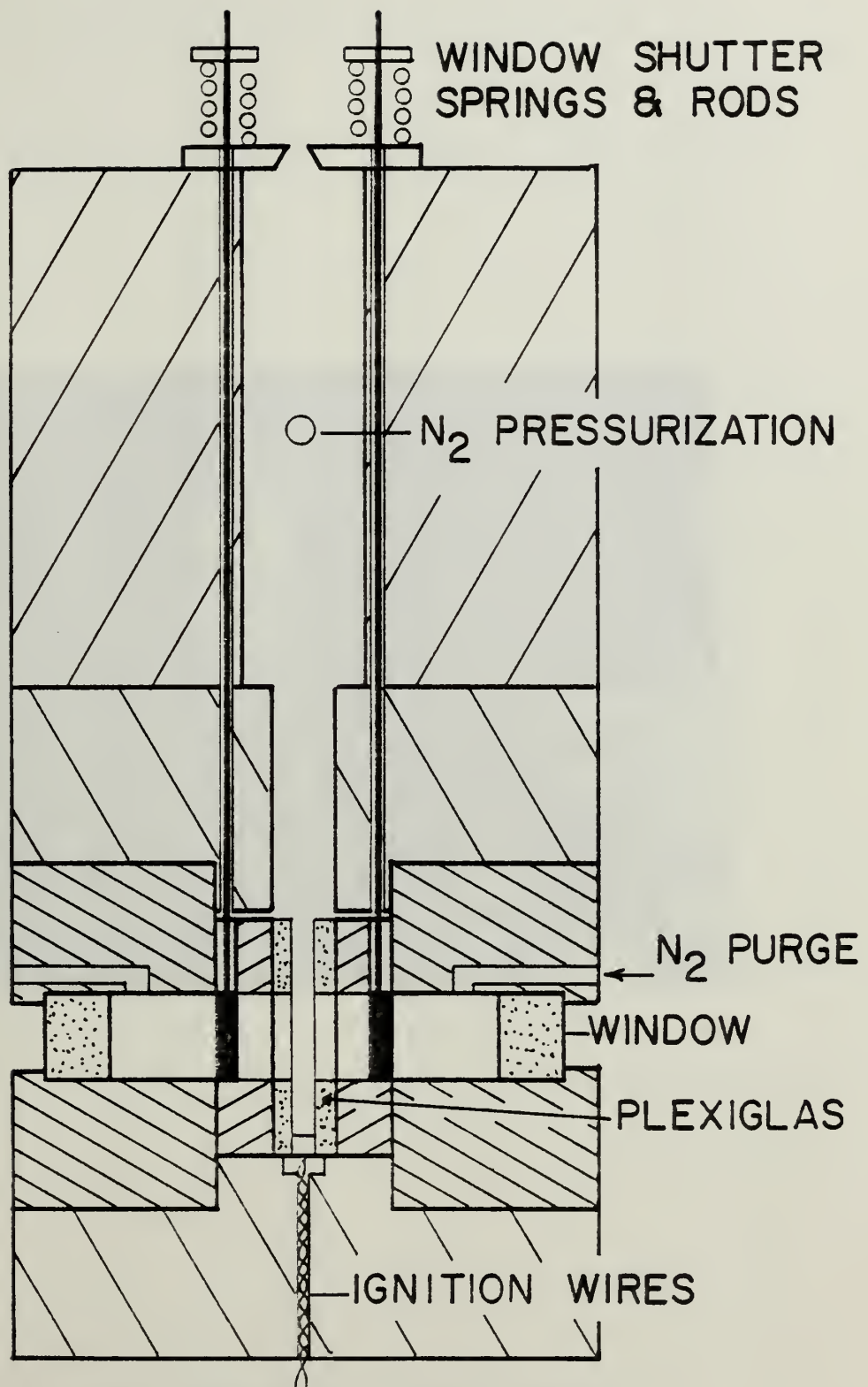


Figure 6. Motor Line Drawing, Side View

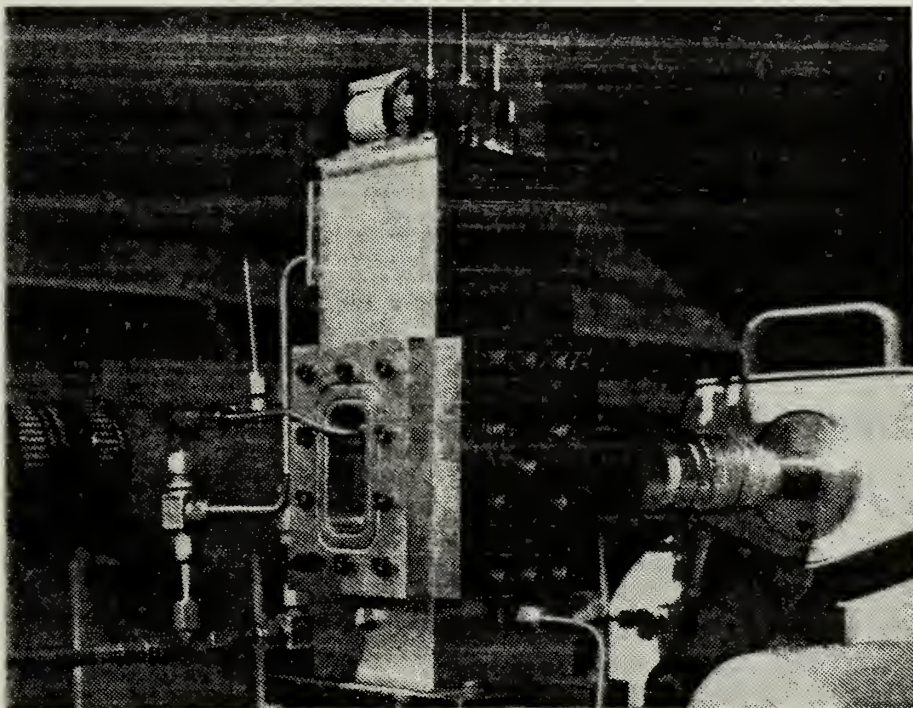


Figure 7. High Speed Motion Picture Apparatus

TABLE I

Propellant Composition and Initial Aluminum Particle Size

| Propellant Designation | Binder % Weight | Oxidizer % Weight | Metal % Weight | Mean Metal Diameter, micro |
|------------------------|-----------------|-------------------|------------------------------------|----------------------------|
| WGS-5A | HTPB 12 | AP 83 | Al 5 | 75-88 |
| WGS-6A | HTPB 12 | AP 83 | Al 5 | 45-62 |
| WGS-7A | HTPB 12 | AP 83 | Al 5 | 23-27 |
| NWC-1 | HTPB 12 | AP 87 | Al ₂ O ₃ 1.0 | 8 |
| NWC-2 | HTPB 12 | AP 87.5 | Al ₂ O ₃ 0.5 | 8 |

Samples were rough-cut to approximate size, then hand rubbed to final dimensions. This rubbing also served to smooth any saw marks caused by the rough cut and removed any loose propellant which might have caused debonding of the inhibitor. Initially, quite large opposed propellant slabs (2.0 inches long x 0.5 inch web x 0.1875 inch thick) were used. These large slabs allowed the propellant to provide its own pressurizations; however, the smoke produced proved to be too dense for the laser to penetrate. Eventually, with the addition of a secondary nitrogen pressurization chamber and the elimination of the opposing slab, a final propellant dimension of approximately 1.13 inches x 0.5 inch web x 0.025 inch thick was employed. The propellant slab is represented in Figure 8.

In this configuration, the propellant had to provide only approximately five percent of the desired steady state combustion pressure. The major portion of the chamber pressurization was provided by the

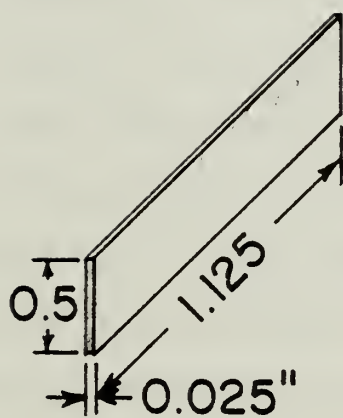


Figure 8. Propellant Slab with Inhibitor

nitrogen introduced downstream of the propellant. The sample was epoxy bonded to a propellant support base and inhibited with a very thin coat of General Electric Hi-Temp Gasket (Red RTV), then allowed to cure for twenty-four hours. Other schemes examined to inhibit the propellant were to moisten the surfaces to be inhibited with water to dissolve a portion of the ammonium perchlorate (oxidizer) or to apply a thin coat of epoxy resin to the surface.

The system was prepared for firing by installing the propellant and the window blocks into the motor. The plexiglas spacers were shimmed as necessary to ensure contact with the propellant and then the steel L-shaped spacer was attached. The ignitor charge was inserted into the gap in the steel L-shaped spacer and then primed with a small amount of black powder (and cellulose based glue dissolved in acetone) deposited on an electric ignition wire. After completing the assembly of the motor, the necessary electrical connections were made, the holocamera was set in place, and the laser was prepared for firing.

A firing sequence consisted of the following:

1. Check electrical connections and plug ignition wires into firing circuit
2. Charge the laser to its firing voltage
3. Start the Visicorder
4. Pressurize the motor with nitrogen to about 475 psi for a desired test pressure of 500 psi
5. Fire the ignition switch
6. Propellant ignites and combustor pressure builds
7. Pressure sensor monitors combustor pressure and starts time delay at preset pressure
8. Time delay expires and energizes window shutter solenoid

9. Solenoid retracts and releases window shutters
10. Window shutters open
11. Window shutters trip laser fire microswitch, opening holocamera shutter and laser fires.

The sequence of events 3-11 usually took less than 4 seconds to complete.

After the film plate was exposed, it was removed from the plate holder in a dark room and processed as follows:

1. Kodak D-19 developer was used as the processing agent. The plate was bathed from 1 to 3 minutes and inspected periodically under a Kodak safelight. When a satisfactory "image" had formed, the plate was removed to a fresh water rinse then placed in the fixer.
2. Kodak "Rapid Fix" was used to set the image. Processing time was 5 to 7 minutes.
3. After fixing, a 15 minute fresh water wash was conducted followed by 30 seconds in Kodak "Photo Flo" solution, then the plate was hung to dry.

D. DISCUSSION

An essential feature of the data was that the hologram be taken during a period when the propellant was burning in a steady-state environment so that realistic conditions would be more closely simulated. The determination of when steady-state conditions existed was noted from previous pressure versus time plots and used to set the pressure sensor and time delay for each firing. The goal was to have the pressure switch activate at a preset point on the rising pressure gradient such that the time delay resulted in the hologram being taken during steady state pressure.

Although the intensity of flame envelopes is reduced by the use of appropriate optical filters, the thermal gradients which also encase each particle become evident in the hologram. These "thermal cells" are manifested by density discontinuities around each particle and cause the beam to refract (similar to a schlieren effect). This problem is minimized by introducing an optical diffuser into the scene beam prior to its passing through the motor. The result is that the beam is scattered uniformly, and the thermal cell images are averaged. However, the introduction of the glass diffuser results in images characterized by speckle (optical noise generated by interference sources in the beam path). Removal of this speckle is essential to the preservation of the resolution capability of the system.

The speckle removal technique employed was a refinement of that used by Karagounis [Ref. 12], and originally used by Wuerker and Briones [Ref 13]. A rotating mylar disk was placed at the focal plane of the microscope and the reconstructed image was then viewed on the mylar sheet. In place of the simple sliding table on which the spinning disk was mounted in the original apparatus used by Karagounis, a vernier transit was employed. This system allowed the precise positioning of the disk in the focal plane of the microscope and ensured that it remained as positioned.

Each type of propellant requires extensive testing to determine the proper reference beam filtering. A ratio of approximately 5:1 (reference beam:scene beam) is required for a high resolution hologram. The proper ratio was determined by conducting tests during which the scene beam and reference beam were only partially overlapped. When the plate was developed, an estimation of the required neutral density filter for the reference beam could be made.

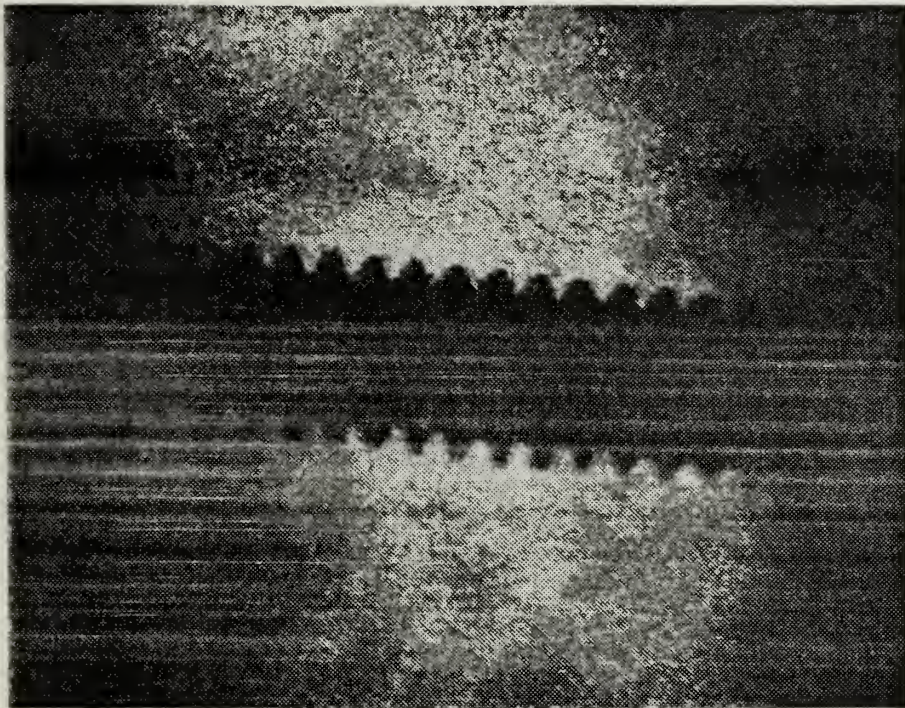
Extensive testing was required to develop the final technique due to the presence of large quantities of smoke in the combustor. Overcoming the smoke density was the single most vexing problem of this investigation. The sources of the smoke were from the burning propellant, the burning black powder ignitor, and the burning inhibitor. A solution to the problem required (1) very thin (0.025 in.) propellant slabs, (2) elimination of the opposed slab, (3) very thin inhibitor coatings in solid contact with the plexiglas spacers, and (4) increased laser power density.

Laser power density was increased by changing the optics in the beam expander. The result was a smaller diameter beam with the capability to penetrate denser smoke.

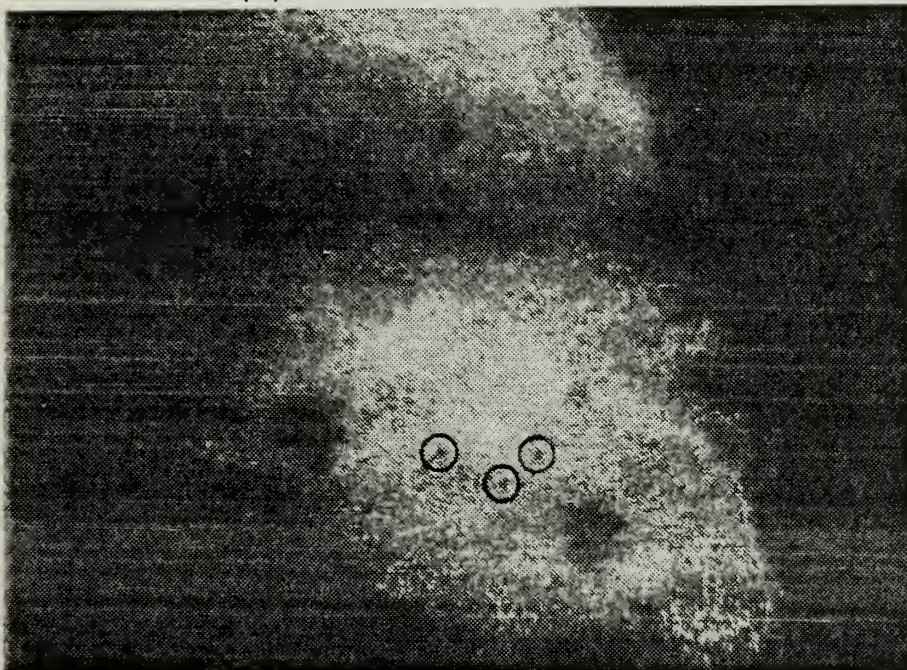
IV. RESULTS AND RECOMMENDATIONS

A technique to obtain holographic recordings of the combustion process in a two-dimensional solid propellant rocket motor was developed during this investigation. A hologram of WGS-6A propellant was recorded at a steady state pressure of 420 psig in a cross-flow environment (of approximately 1.5 m/sec) with a laser pulse length of 50 nanoseconds duration. Figure 9(a) is a reconstructed hologram of a 0-80 screw which was placed in the window hole to provide a scale for particle size determination. The threads are 317.5 microns from peak-to-peak. Figure 9(b) is a reconstructed hologram of the burning propellant. The vertical propellant surface is on the right side and the flow direction is upwards. The large particles to the left are of unknown origin, but may be particles of material from the ignitor or the inhibitor. If the relatively massive particles to the left are discounted, no appreciable agglomeration of aluminum is evident at this stage of the combustion process.

Greater laser power may be required to provide sufficient power densities to penetrate the smoke in the motor for propellants containing more aluminum or for thicker slabs. In addition, increased scene beam light intensity on the hologram could be obtained by enlarging the exit window of the motor. This would allow more laser light from inside the motor to fall on the film plate, and would effectively increase the output power density without modification to the input beam.



(a) 0-80 Screw



(b) WGS-6A Propellant

Figure 9. Photograph of Reconstructed Hologram of WGS-6A

Motion pictures were taken of WGS-6A at 1000 and 4000 frames per second and of WGS-7A at 4000 frames per second with both propellants at a steady state pressure of 420 psig. Representative frames from each of these motion pictures are presented as Figures 10 and 11. Observation of the particle paths in each of these motion pictures provides visual verification of cross-flow conditions. As would be expected, those particles with the smaller initial sizes tended to track the cross-flow velocity field more closely than the larger particles.

Areas of this investigation which require further consideration are the reliability of the motor ignition system, the repeatability of the pressure-time trace, and the timing of the cinematic/holographic observation.

The current motor ignition system does not provide consistent run to run results. The ignitor must generate the conditions necessary for the propellant to ignite uniformly and to reach steady state pressure in a very short period of time. Minimizing the time to reach steady state minimizes the smoke density in the combustor when the observation is made. It is the reduction of the smoke to a penetrable density that makes the holographic technique possible.

Modifications to improve the ignition system reliability might include changing the ignitor charge shape or position to achieve a better hot particle spray pattern across the propellant surface.

Improving the repeatability of the pressure-time conditions may be had by exercising proper quality control during the motor assembly process. It is essential that the combustor be effectively sealed and the propellant faces protected from the high temperature combustion products.



(a) 1000 Frames Per Second

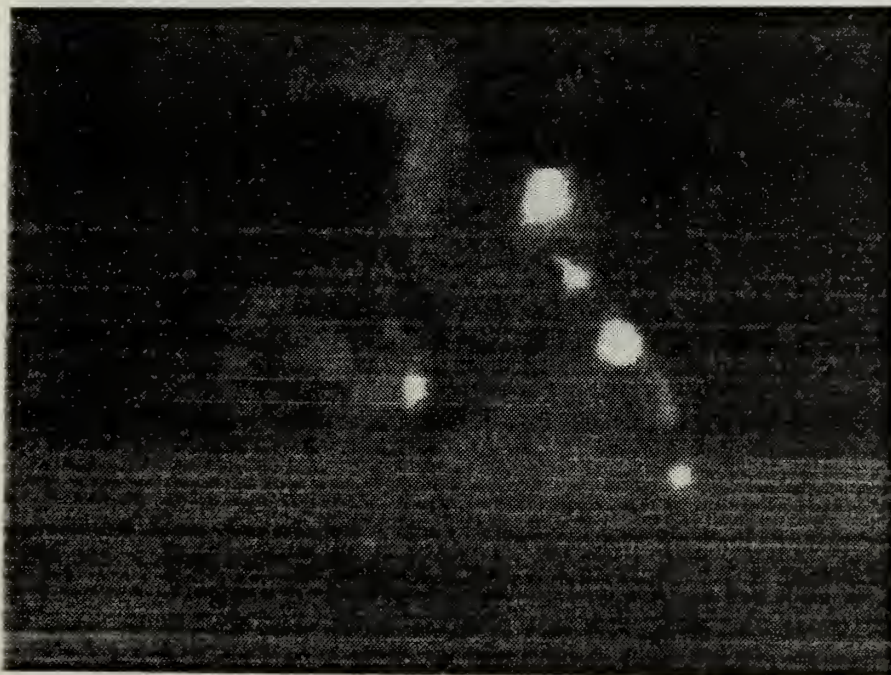


Figure 10. Motion Picture Frame of WGS-6A

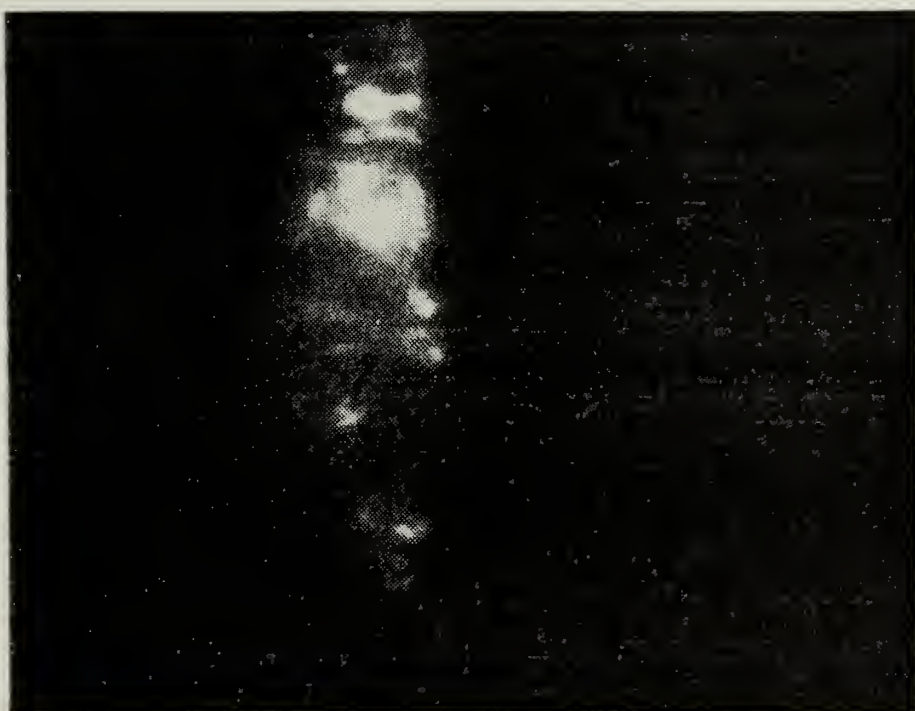


Figure 11. Motion Picture Frame of WGS-7A

This is accomplished by carefully installing the plexiglas spacers to ensure physical contact between the propellant and the plexiglas so that the steel L-shaped spacer (opposing the propellant slab) seals the combustor.

Improvements in the timing of observations to coincide with steady state conditions will come with more experience in operating the system.

With firing preparations and motor assembly requiring six hours or more, solutions to these problems are necessary if holograms and motion pictures are to be obtained in a timely manner. The system as designed and operated will produce useable holograms; however, extreme care must be exercised in system preparation to ensure that accurate data is generated on each firing run.

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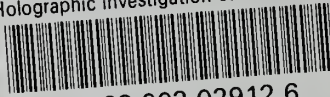
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